ANALYST:		VPDES NO	

Parameter: Oil & Grease
Method: HEM
09/05

METHOD OF ANALYSIS:

	EPA Method 1664 Revision A

		Y	N
	COLLECTION		
1)	Are glass bottles with Teflon (PTFE) lined screw caps used for sample collection? [6.1.1]		
2)	Have collection bottles been properly cleaned? NOTE: If blanks demonstrate that the collection containers and lids are clean, the cleaning preparation may be eliminated. (Bottles - detergent washed, rinsed with water, and then either solvent rinsed or baked at 200-250°C for 1 hr prior to use? Liners for screw caps - detergent washed, rinsed with water and solvent rinsed and baked at 110-200°C for 1 hr prior to use?) [6.1.2]		
3)	Is enough headspace left at top of collection bottle to allow for pouring without loss of sample?		
4)	Are samples adjusted to a pH of <2 with HCl or H_2SO_4 and refrigerated at 0-4°C? [8.1.1 & 8.4]		
5)	Are samples analyzed within 28 days of the date and time of collection? (8.5)		
	QUALITY CONTROL		
6)	Have MDLs been determined? [9.2.1]		
7)	Has initial precision and recovery been demonstrated? [Analyze 4 precision and recovery (PAR) standards and compute average percent recovery (X) and standard deviation of % recovery (s). Results must meet Acceptance Criteria -- HEM (s) ±11%; (X) 83-101% [9.2.2]		
8)	Are matrix spikes performed on 5% of samples from a given discharge/waste stream by adding the spiking solution to the sample container? [9.3]		
9)	Are spike recoveries within the acceptable range? HEM - 78-114% [9.3.4]		
10)	Are laboratory reagent water blanks analyzed to demonstrate freedom from contamination? [9.4]		
11)	Is balance calibration verified using Class 2 weights (formerly Class S for weights 1 mg to 10 g) at beginning and end of each day of analysis? NOTE: Calibration must be within 10% at 2 mg and 0.5% at 1000 mg. [9.5 & 10.0] **NOTE:** *Class S weights have been replaced by Class 1 for measurements of 20g and above, and Class 2 for weights 10 g or less.*		
12)	Is a PAR standard which is added to a sample container, analyzed with each batch? Results must meet Acceptance Criteria -- HEM (X) 78-114% [9.6]		
13)	Is level of standard marked on container after each day of use and then reconstituted with acetone prior to subsequent use? [7.10.2 & 7.10.3]		
14)	Is a quality control sample (QCS), from source different than the standard used, routinely analyzed? [9.7]		
	PROCEDURE		
15)	Are samples brought to room temperature prior to analyses? [11.1.1]		
16)	Is sample volume determined by either marking water meniscus or weighing bottle for later determination? [11.1.4]		
17)	Is pH of <2 verified by dipping a glass rod into sample; touching rod on pH paper; then rinsing rod with hexane over the sample, thus including the rinsate in the sample extraction? [11.2.1]		
18)	Is all glassware solvent rinsed or baked at 105-115°C after cleaning? [4.3]		

		Y	N
19)	Are cleaned boiling flasks containing 3-5 boiling chips baked at 105-115°C for a minimum of 2 hrs. prior to being placed in desiccator for cooling and determining the tare weight? [11.3.1]		
	Separatory Funnel Extraction		
20)	Are 30 mLs of n-hexane added to sample bottle, which is then sealed with original cap; shaken to rinse all interior surfaces; and then poured into the separatory funnel (2 L funnel fitted with a Teflon stopcock)? [11.3.3 - 6.4.3]		
21)	Is sample extracted by vigorously shaking the separatory funnel for 2 mins.? [11.3.4]		
22)	Are layers allowed to separate for a minimum of 10 mins. before draining the lower layer into the original sample container? [11.3.5 & 11.3.6]		
23)	Is the tared weight of the distilling flask recorded? [Permit]		
24)	Is solvent layer drained through a Whatman 40 (or equivalent) filter holding approximately 10 g of pre-rinsed sodium sulfate ($NaSO_4$) into a tared boiling flask? [11.3.8]		
25)	Are 3 extractions performed on each sample? [11.3.9]		
26)	Is a small amount of n-hexane drained from separatory funnel with each extraction? [11.3.6]		
27)	Are the separatory funnel tip, filter paper, and funnel rinsed with 2-3 small portions of n-hexane which is then added to the flask? [11.3.10]		
	Solid Phase Extraction		
28)	Is there a SOP available for SPE? [Permit]		
29)	Is sample bottle rinsed several times using n-hexane with rinsate being added to the filter? [11.3.3]		
30)	Is filter kept moist from time of conditioning until after sample is filtered? [Permit]		
31)	Elution a) Is sample eluted with several aliquots of n-hexane? [11.3]		
	b) Is a small amount of each n-hexane rinse pulled through filter with remaining solvent held in filter for a minimum of 2 mins. prior to starting next rinse? [Permit]		
	c) Are sides of reservoir above filter rinsed with n-hexane? [Permit]		
32)	Are eluents passed through approximately 10 g of pre-rinsed sodium sulfate ($NaSO_4$) into a tared boiling flask with collection vessel rinsate added to flask also? [11.3.8]		
	Evaporation		
33)	Is solvent collected during the evaporation process for reuse? [11.4.1]		
34)	Is temperature of water bath or steam bath adjusted to allow concentration to be completed in 30 mins.? Approx. 85°C [11.4.1]		
35)	Is the sample allowed to distill until the flask appears dry or distillation head reaches 70°C? [11.4.2]		
36)	Following evaporation, is the flask swept of solvent fumes with a vacuum for 15 secs.? [11.4.2]		
37)	Is the flask wiped clean of moisture and fingerprints, dried in oven at 70 ± 2°C for 30 min., and then placed in a desiccator for 30 mins. minimum prior to weighing? [11.4.2 - 11.4.4]		
38)	Was drying cycle repeated until weight loss was < 4% of previous wt. or < 0.5 mg, whichever was less? [11.4.4]		
39)	Is weight of the distilling flask and residue recorded? [11.4.4]		

		Y	N
40)	Is weight of the residue calculated and recorded? [11.4.4.1]		
41)	Is the volume of the original sample determined and recorded? [11.4.5]		
42)	Is calculation for the concentration of HEM (oil and grease) correct and shown on the bench sheet? [12.1]		

$$HEM(mg/L) = \frac{W_h(mg)}{V_s(L)}$$

where :
W_h = Weight of extractable material
V_s = Sample volume

PROBLEMS: